

[Field of the Invention] This invention Organic polymeric materials, such as a woody material and plastics, ceramics, it is related with the sustained-release material which made porous materials, such as charges of non-equipment, such as glass, support an agreeable odor or the scent component for deodorization, medical properties, the object for insect control or the component for insect killing, a fertilizer component, and the other [0001]

[Detailed Description of the Invention]

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[Claim 8] The manufacture procedure of a sustained-release material according to claim 3 to 7 which is the materials with which gradual release component content materials to 6 that supercritical fluid or subcritical fluid is carbon dioxide, the nitrous oxide, trifluoromethane, or two or more sorts of mixtures of them.

[Claim 7] The manufacture procedure of a sustained-release material according to claim 3 materials, such as a woody material and plastics, ceramics, and glass, or these composite to 5 that porous materials are charges of non-equipment, such as organic polymeric [Claim 6] The manufacture procedure of a sustained-release material according to claim 3 moisture, contacts supercritical fluid or subcritical fluid.

[Claim 5] The manufacture procedure of a sustained-release material according to claim 4 which is caused especially or is made by vacuum drying or lyophilization that elimination of impurities which have adhered in porous one of a porous material, such as oil and [Claim 4] Impurities which have adhered in porous one of a porous material, such as oil and moisture, are removed. And the manufacture procedure of the sustained-release [Claim 3] The manufacture procedure of the sustained-release material characterized by manufacturing the sustained-release material with which the gradual release component content materials, making the extracted gradual release component support in porous one of said porous material, and manufacturing sustained-release material by contacting supercritical fluid or subcritical fluid.

[Claim 2] Sustained-release material according to claim 1 whose porous materials are charges of non-equipment, such as organic polymeric materials, such as a woody material and plastics, ceramics, and glass, or these composite materials.

[Claim 1] Sustained-release material characterized by having made the gradual release component support in porous one of a porous material, and constituting it through supercritical fluid or subcritical fluid.

[Claim(s)]

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## CLAIM + DETAILED DESCRIPTION

[0002] components used so that it may do sustained-release so, and the manufacture procedure of the sustained-release material.

[0002] Description of the Prior Art] As technology of making porous materials, such as charges of non-equipment, such as organic polymeric materials, such as a woody material and plastics, ceramics, and glass, supporting a scent component etc., conventionally The extractives or the solution sinking-in method which was compounded artificially and which is fragrant, and applies a component to material, or is infiltrated extracted from the materials containing a scent component is mainly adopted. Moreover, adhesion of the scent component by vacuum impregnation is also performed.

[0011] As a porous material, charges of non-equipment, such as organic polymers materials, such as a woody material and plastics, ceramics, and glass, or these composite materials are used.

[0012] Moreover, not only the material of the narrow sense before being produced commercially but the product itself is contained in a porous material. Therefore, it is wooden products, a product made from plastics, etc., and various kinds of products after molding are also contained.

[0013] Therefore, it is contained also when it seems that supercritical fluid or subcritical fluid is made to permeate the interstice of the textile fabrics created for the details of a product (for example, Buddha statue) with which fine sculpture was given, fiber, or its fiber etc. with a gradual release component.

[0014] Moreover, the nitrous oxide, trifluoromethane, or two or more sorts of mixtures of them are used, for example, for the object for agreeable odors or the scent component for deodorization, for insect killing as gradual release component materials, the materials containing a fertilizer component, etc. are used.

[0015] Furthermore, the materials which contain the component for the materials which contain the object for agreeable odors or the scent component for deodorization, for example, the materials containing medicinal properties, the object for insect control, or insect killer, the materials containing the component for the materials which do so here.

[0016] Gradual release components are various kinds of components, such as the above scent components, and mean the component used so that it may make sustained-release used for carrying out the invention] The embodiment of this invention is hereafter explained according to Drawings.

[0017] [0018] (Embodiment 1) Drawing 1 shows the outline block diagram of the equipment used for manufacturing of the scent component support woody material as one embodiment.

[0019] The equipment used for the manufacture procedure of the scent component support woody material of this embodiment possesses the hyperbaric pressure container 1 which accommodates the scent component materials 9 and the woody material 8 to which the scent component materials are made to adhere, as shown in drawing 1.

[0020] This hyperbaric pressure container 1 is a product made from stainless steel, and as shown in this figure, it consists of a package body 2 and a lid 3.

[0021] The manufacturing installation of the scent component support woody material of this embodiment possesses the cylinder 4 is a cylinder for storing the fluid which forms supercritical fluid or manometer 6, and the back pressure valve 7 further.

[0022] A cylinder 4 is a cylinder for storing the fluid which forms supercritical fluid or manometer 6, and carbon dioxide is used by this emboidment as fluid.

[0023] The hyperbaric pressure pump 5 is a pump for supplying the fluid in said cylinder 4 to said hyperbaric pressure container 1, and the tension of the hyperbaric pressure pump 5 is measured with said manometer 6.

[0024] A back pressure valve 7 is for being able to make it open and close by predetermined tension, and keeping operating pressure power constant to a predetermined value.

[0025] Furthermore, the thinning for which a back pressure valve 7 is opened completely and decompressed -- \*\*\* -- depression separation of the supercritical fluid will be carried out from the hyperbaric pressure container I.

[0026] In addition, the piping part (the diagram is illustrating) etc. possesses in the manufacturing installation of the scent component support woody material of this manufacturer wood material is explained using such equipment.

[0027] Next, the embodiment of a procedure which manufactures a scent component embodiment.

[0028] First, the Chinese medicine galenicals as the woody material 8 which is the target of scent component adhesion, and scent component materials 9 are enclosed in the hyperbaric pressure container I is set as the target temperature, and further, after setting the release tension of a back pressure valve 7 as the target tension, carbon dioxide is supplied to the hyperbaric pressure container I using the hyperbaric pressure pump 5.

[0029] Next, the constant temperature bath (not shown) which has inserted in the hyperbaric pressure container I.

[0030] Carbon dioxide serves as supercritical fluid under the temperature of 31.1 degrees Celsius a cylinder 4.

[0031] the predetermined time neglect after the temperature and tension in the hyperbaric pressure container I reach a predetermined value -- it carries out. A scent component is pressurized by the woody material 8.

[0032] If this is explained in detail, first, with supercritical carbon dioxide, from scent component materials, a scent component will be extracted, next it will be fragrant with the woody material 8, after a certain amount of impurities are removed from a porous remain in porous one of the woody material 8, when supercritical carbon dioxide contacts the woody material 8, it is fragrant to that porous inside, and a component permeates.

[0033] In this case, although impurities, such as resin, oil like the sap, and moisture,

[0034] After are fragrant to the porous deep part of the woody material 8 and making a gasous status due to decompression, and diffusion elimination of the supercritical component permeate by [predetermined] carrying out time neglect, a back pressure valve 7 is changed into release status.

[0035] By this, a pass will be in depression status, supercritical carbon dioxide returns to carbon dioxide is automatically carried out from a woody material.

[0036] An adsorption supplement is carried out inside porous one and one of these and a scent component remains to a woody material.

[0037] Thus, although the scent component support woody material with which the scent component was supported will be manufactured. Since carbon dioxide serves as supercritical fluid as mentioned above under the temperature of 31.1 degrees C (critical temperature), the temperature more than tension 73 atm (critical pressure), and the conditions of tension, it can set temperature as low temperature comparatively, therefore can prevent deterioration by the heat of the woody material 8 and the scent component materials 9.

[0038] (Embodyment 2) Drawing 2 shows the outline block diagram of the equipment used for manufacturing of the scent component adhesion woody material as other embodyments.

[0039] Two hyperbaric pressure containers possess in the equipment used for the manufacture procedure of this embodiment.

[0040] In one hyperbaric pressure container 1a, it is fragrant, the component materials 9 are accommodated, and the woody material 8 is accommodated in the hyperbaric pressure container 1b of another side.

[0041] The circulation way 11 which circulates the supercritical fluid other than a pass 10 from the fluid stagnation way 11 to a back pressure valve 7 is formed.

[0042] The pump 12 is formed in this circulation way 11.

[0043] The point that the cylinder 4, the hyperbaric pressure pump 5, the manometer 6, and the back pressure valve 7 possess is the same as the above-mentioned embodiment 1.

[0044] The valve 13 is formed between the hyperbaric pressure pump 5 in a pass 10, and the manometer 6.

[0045] Moreover, valves 16 and 17 are formed in one outward trip 14 and return trip 15 the manometer 6.

[0046] Furthermore, valves 20 and 21 are formed in the outward trip 18 and return trip 19 to the hyperbaric pressure container 1a, respectively.

[0047] Furthermore, a valve 22 is formed in the outward trip 14 to one hyperbaric pressure container 1a, and the pass 10 between return trips 15, and the valve 23 is formed in the outward trip 18 to the hyperbaric pressure container 1b of another side, and the pass 10 between trips 19 to the hyperbaric pressure container 1a, respectively.

[0048] Furthermore, two valves 24 and 25 are formed in the circulation way 11.

[0049] Next, the manufacture procedure of the scent component support woody material of this embodiment is explained using the above equipment.

[0050] First, the impurities which remain in porous one of the woody material 8 are removed.

[0051] In this drying stage, while changing valves 13, 22, 20, and 21 into the status of "\*,", valves 16, 17, 23, 24, and 25 are changed into the status of "\*\*". Moreover, a back pressure valve 7 is set up to open by predetermined tension.

[0052] While the outward trip 14 to the hyperbaric pressure container 1a with which the pressure value 7 is set up to open by predetermined tension.

[0053] After carbon dioxide is powdered in to the hyperbaric pressure container 1b, by pressurizing flow way 11 is closed, and carbon dioxide is supplied to the hyperbaric pressure container 1b with which the woody material 8 was accommodated by it.

[0054] Next, operation of removing the powdered-in supercritical carbon dioxide from the material 8.

[0055] At this aspiration process, although the opening-and-closing status of each valve in the above-mentioned drying stage is almost the same, it is different with the point which changes a valve 13 into the status of "\*\*". Moreover, a back pressure valve 7 is changed into the status of full open.

[0056] Supply of the carbon dioxide from a cylinder 4 stops, and the supercritical carbon dioxide in the hyperbaric pressure container 1b is discharged by the part outside the system from a back pressure valve 7 by this.

[0057] As a result, it means that the supercritical carbon dioxide in the hyperbaric pressure container 1b from a return trip 19, and is further discharged out of a hyperbaric pressure container 1b is discharged by the part outside the dioxygen 9 were accommodated, a scent component is supplied to the hyperbaric pressure container 1b with supply of oxygen 9.

[0058] Next, while supplying carbon dioxide to the hyperbaric pressure container 1a with pressure container 1b was removed.

[0059] At this extraction supply process, while changing valves 13, 16, 17, 20, and 21 into the status of “\*\*”, valves 22, 23, 24, and 25 are changed into the status of “\*\*”.

[0060] From a cylinder 4, carbon dioxide is supplied to the hyperbaric pressure container 1a by this, and is fragrant with this, it is fragrant from the hyperbaric pressure container 1b with which the scent component dioxygen 9 was stored with supercritical carbon dioxide.

[0061] Next, the scent component to the woody material 8 is supported.

[0062] At this support process, while changing valves 16, 17, 20, 21, 24, and 25 into the status of “\*\*”, valves 13, 22, and 23 are changed into the status of “\*\*”. Moreover, a setup of a back pressure valve 7 is the same as a last process.

[0063] By this, a scent component circulates through the circulating flow way 11, the hyperbaric pressure container 1a, and the hyperbaric pressure container 1b with supercritical carbon dioxide, without newly supplying carbon dioxide from a cylinder 4.

[0064] As a result, the extracted scent component will be supported by the woody material 8. The particles of the above components will be supported in porous one of the woody material 8 from which the above mentioned dryring stage showed to drawing 3.

[0065] Then, the woody material 8 with which the scent component was supported is picked out from the woody material 8 with which the scent component is completed.

[0066] Manufacture of the scent component support woody material with which the scent component was supported as mentioned above is completed.

[0067] Also in this embodiment, temperature can be comparatively set as low 1, and deterioration by the heat of the woody material 8 and the scent component further by using supercritical carbon dioxide like the above-mentioned embodiment [0068]. Since two hyperbaric pressure containers of the hyperbaric pressure container 1a which accommodates the scent component materials 9, and the hyperbaric pressure container 1b which accommodates the scent component materials 9, and the hyperbaric pressure container 1b which is made to provide further in this embodiment, operation of making supercritical carbon dioxide removing pressure container 1b which accommodates the woody material 8 were made to provide impurities, such as a resinous principle in porous one of it having been moisture, first. After the separate container could perform operation of it having been extracting, therefore making impurities, such as a resinous principle in porous one of the woody material 8, and removing woody material 8, and moisture, remove certainly. Since the carbon dioxide by which fragrance from the scent component container 9, and making a component

[0069] (Other embodiments) In addition, by each above-mentioned embodiment, in order are fragrant from scented components and to extract a component, supercritical carbon dioxide was used, it is also possible to use supercritical fluid or subcritical fluid other than carbon dioxide. Furthermore, in the above-mentioned embodiment, although supercritical carbon dioxide was used, it is also possible not only this but to use the charge of a ceramic material, and it is also possible to use an organic polymeric material. Moreover, as a form of material, the solid of a globular shape, tabular, and other form, fiber, textile fabrics, and paper can be used.

[0070] Furthermore, in the above-mentioned embodiment, although the woody material to use what is called supercritical carbon dioxide near it.

[0071] Furthermore, in the above-mentioned embodiment, although supercritical carbon solvents, such as lower alcohol, such as methanol, ethanol, and propanol, or acetone, and chloroform, to supercritical fluid.

[0072] Furthermore, the kind of scented component is not limited to the above-mentioned embodiment, either, and the kind is not asked. Moreover, it is also possible to use used.

[0073] Furthermore, in the above-mentioned embodiment, such as globular components which vaporizes in gradual release from the target porous material in short example, a medicine component, an insecticide, an insecticide, and manure. The components, such as gradual release components other than a scented component, for example, a medicine component, an insecticide, an insecticide, and manure. The volume of 50ml.

[0074] (Work example 1) 10.30g of wooden roses were prepared as a woody material, 5.2g of ten kinds of incense of \*\*\*\*\* were prepared as scented component materials.

[0075] In addition, said ten kinds of incense consist of a sandalwood, a clove, the chinamom, Hedychium spicatum, \*\*\*\*\*\*, spikenard, star anise fruit, borneol, and aromatic tree sedimentation.

[0076] Next, while setting temperature as 50 degrees C, after setting tension as 20MPa and supplying carbon dioxide to a hyperbaric pressure cell, it changed into the status of supercritical carbon dioxide, this status was neglected for 3 hours, and the scented elimination was carried out from the woody material.

[0077] After scented component support, by releasing a back pressure valve and reducing tension, supercritical carbon dioxide was returned to gaseous status, and diffusion be obtained as a result of the experiment.

[0078] The wooden roses with which the scented component was supported were able to [0079] (Work example 2) As a woody material, 36.34g wooden Buddha statues were prepared, 18.19g of ten kinds of incense of \*\*\*\*\* were prepared as scented component materials, and it enclosed with the hyperbaric pressure cell made from stainless steel with a content volume of 300ml.

[0080] In addition, since the operating condition of this example is the same as said work

[0081] Furthermore, in this invention, since it was fragile, such as organic has permeated deeply, the scented effect can be made to maintain for a long period of time.

[0082] (Example of an examination) In the example of an exam, temperature, tension, and drying time were examined as a variable factor.

[0083] First, weighing of about 12g was carried out, and the bead was enclosed in the liquid-sealing speed, the temperature of the constant temperature bath was set up.

[0084] Then, carbon dioxide was circulated, it circulated and elimination of the impurities which remain in a rosary was started. After predetermined time progress, it decompressed and the rosary was taken out.

[0085] Using the Shimadzu make and an AEG-45SM type electronic balance, the obtained sample measured weight and measured the weight before and behind desiccation. The rate of weight loss (%) =  $\frac{\text{After [Materials g]-desiccation [g]} - \text{Materials [g]}}{\text{Materials [g]}}$

[0086] Rate of weight loss [%] =  $\frac{\text{After [Materials g]-desiccation [g]} - \text{Materials [g]}}{\text{Materials [g]}}$

[0087] As a result of the examination, as shown in the graph of drawing 4, the rate of weight loss was increased with time. Moreover, in the condition than drying time 2 hour that it does not increase so much. But it turned out in more than drying time 4 hours that it increases with time, but it turned out in the rate of weight loss in the graph of drawing 5, it turned out that nearly 40 degrees C is made into a peak (maximum), and there is a trend for the rate of weight loss to fall in the temperature range of the both sides. This is considered to originate in the dissolution of carbon dioxide being so high that temperature being low under the same tension conditions, and the effect of dissolving an organic matter being high in supercritical conditions, On the 25-degree C conditions below the critical temperature, since carbon dioxide is not in supercritical status, even if the density of fluid is high, the dissolution effect of an organic matter and the pass-through effect to wood are considered to be because it to have decreased.

[0088] Furthermore, the rate of weight loss in tension 20MPa, carbon dioxide 3 ml/min, whose temperature is 40 degrees C was brought.

[0089] As shown in the graph of drawing 5, it turned out that nearly 40 degrees C is made into a peak (maximum), and there is a trend for the rate of weight loss to fall in the temperature range of the both sides. This is considered to originate in the dissolution of carbon dioxide being so high that temperature being low under the same tension conditions, and the effect of dissolving an organic matter being high in supercritical conditions, On the 25-degree C conditions below the critical temperature, since carbon dioxide is not in supercritical status, even if the density of fluid is high, the dissolution effect of an organic matter and the pass-through effect to wood are considered to be because it to have decreased.

[0090] (Effect of the Invention) As mentioned above, by contacting supercritical fluid or subcritical fluid in a porous material and scented component materials in this invention It is furthered in a porous material, and a component can be extracted, it can be further fragrant with supercritical materials, and a component can be made to further fragrant whole field, and a component can be made to support not to mention the Taira glide plane by the deep part inside the osculum of a porous material.

[0091] Furthermore, it can be fragrant to the complicated-shaped whole field, and a component can be made to pass-through effect, such as supercritical fluid.

[0092] Furthermore, since it was fragile, such as organic has permeated deeply, the scented effect can be made to maintain for a long period of time.

[0093] Furthermore, in this invention, since it was able to be adhered as a result of an example 1, the explanation is omitted.

solvent, are not used so much, it is not necessary to carry out the drying stage of a product etc. after a scent component support process.

[0094] Furthermore, since waste fluid is not generated in a process, it becomes unnecessary [the process of treatment of waste fluid etc.].

[0095] Furthermore, the scent adhesion process as a secondary process can be carried out to what is already completed as a product, without breaking the form, and a scent can be made to adhere.

[0096] By contacting supercritical fluid or subcritical fluid to a porous material By removing impurities which remain in porous one of a porous material, such as oil and moisture, and content materials after that in extracting a gradual release component from component content materials, making a porous material support the gradual release component content materials, making a porous material support the gradual gradual release component and manufacturing sustained-release material.

Operation of making supercritical carbon dioxide removing impurities, such as oil in pores one of a porous material, and moisture, After being able to perform operation of it having been fragment from scent component content materials, and making a component extracting, at the separate process, therefore making impurities, such as oil in pores one of a porous material, and moisture, remove certainly Since the component by which supercritical extraction was carried out can be made to permeate in porous one of the, there is an effect of the ability to make a scent component support certainly with a woody material.